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Indian Standard

SYNTHETIC SULPHOCHLORINATED FATLIQUORS FOR LEATHER PROCESSING — SPECIFICATION

UDC 675·045·4

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Price Group 2

Tanning Materials and Allied Products Sectional Committee, CHD 018

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Tanning Materials and Allied Products Sectional Committee had been approved by the Chemical Division Council.

Synthetic fatliquors of sulphochlorinated type are considered essential in the recent years for the production of sophisticated leathers, such as light-weight garments, softie uppers, nappas, gloves, upholstery leathers, etc. The specific advantages of synthetic fatliquors are that they do not possess any obnoxious, foul or rancid odour or render spew formation as generally experienced with natural, marine, vegetable or animal oils based fatliquors. They contain higher emulsifer contents and hence are capable of producing fine water emulsions of penetrative type aiding in even distribution of fat in the leather. Further, the emulsions are insensitive to electrolytes and show remarkable stability to acids and salt solutions normally encountered in the tannery practice.

The leathers processed with the synthetic fatliquors are light-weight and light fast. The basic raw materials needed are based on long straight chain hydrocarbons. The hydrocarbons are made polar by subjecting them to sulphochlorination in the presence of UV-light. The alkane sulphonyl chloride based on heavy normal paraffin is a free flowing clear oily liquid, insoluble in water, soluble in organic solvents like alcohol, benzene, etc, with sulphur content of 5·5 to 6·0 percent and chlorine content of 14 to 15 percent. A variety of fatliquors can be prepared by introducing the hydrophillic groups by reacting the alkane sulphonyl chloride with monoethanolamine followed by sulphation, sulphitation, phosphation, etc.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

SYNTHETIC SULPHOCHLORINATED FATLIQUORS FOR LEATHER PROCESSING — SPECIFICATION

1 SCOPE

This standard prescribes requirements, methods of sampling and test for synthetic fatliquors of sulphochlorinated type used for leather processing.

2 REFERENCES

The Indian Standards listed below are necessary adjuncts to this standard:

IS No.	*Title*
1070 : 1992	Reagent grade water — Specification (*third revision*)
1640 : 1960	Glossary of terms relating to hides, skins and leather
4905 : 1968	Methods for random sampling
5741 : 1970	Methods for determination of *p*H

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 1640 : 1960 shall apply.

4 REQUIREMENTS

4.1 Physical Requirements

The product shall be a free flowing, light brown homogenous liquid/paste with anionic charge and shall form spontaneous emulsion in water.

4.2 The product shall be free from rancid or putrefactive odour.

4.3 Ten percent emulsion in water shall show stability to hard water, acids and chrome liquid when tested in accordance with the method prescribed in **A-1**.

4.4 Chemical Requirements

The product shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in **A-2 to A-7**.

4.5 Keeping Properties

The product shall be stable for at least six months from the date of manufacture when stored in original packings. It shall meet the requirements given in **4.1** to **4.4** when tested any time during this period.

Table 1 Chemical Requirements

(*Clause* 4.4)

Sl No.	Characteristics	Requirements	Methods of Test, Ref to Clause in Annex A
(1)	(2)	(3)	(4)
i)	*p*H of the 10 percent solution	6·5 to 7·0	A-2
ii)	Water content, percent by mass, *Max*	40·0	A-3
iii)	Total fatty matter, percent by mass, *Min*	60·0	A-4
iv)	Total sulphur, percent by mass, *Min*	4·0	A-5
v)	Total chlorine, percent by mass, *Min*	22·0	A-6
vi)	Total ash, percent by mass, *Max*	4·0	A-7

5 PACKING AND MARKING

5.1 Packing

The product shall be packed in airtight containers which shall not develop any rust or deterioration of the product due to presence of water in the medium or any of the ingredients used in the formulation.

5.2 Marking

The containers shall be marked with the following particulars:

a) Name of the product;
b) Name of the manufacturer or its trade-mark, if any;
c) Net content in litres;
d) Date of manufacture; and
e) Batch/Code number.

6 SAMPLING

6.1 Lot

In a consignment, all the containers of synthetic fatliquor for leather processing from the same batch manufactured under uniform conditions shall be grouped together to constitute a lot. Each lot shall be separately tested for various requirements of this specification.

6.2 Scale of Sampling

The number of containers to be selected depends on the size of the lot and shall be in accordance with Table 2. The containers shall be selected by the methods of random selection as given in IS 4905 : 1968.

Table 2 Scale of Sampling

(*Clause* 6.2)

Lot Size (No. of Containers) (1)	Sample Size (*n*) (2)
Up to 50	3
51 to 100	4
101 to 150	5
151 and above	7

6.3 Sufficient quantity of fatliquors from each of the selected containers shall be withdrawn with the help of a suitable sampling implement and be stored separately. One test shall be conducted for lot size up to 100 and two tests shall be conducted for the lot size more than 100 containers for each of the characteristics, namely, *p*H and total fatty matter. After this a composite samples shall be prepared out of the material from individual samples and this composite sample shall be tested for the requirements for stability to hard water, acids and chrome liquid; water content; total combined sulphates; nitrogen content and total ash content. The keeping quality shall be tested by the manufacturer at his end and the corresponding reocords shall be maintained.

6.4 No failure shall occur in the tests conducted above if the lot is to be considered conforming to the requirements of the specification.

7 QUALITY OF REAGENTS

Unless specified otherwise pure chemicals and distilled water (*see* IS 1070 : 1992) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

(*Clause* 4.3, and *Table* 1)

METHODS OF TEST FOR SYNTHETIC SULPHOCHLORINATED FATLIQUORS

A-1 TEST FOR STABILITY

A-1.1 The stability of the emulsion is tested by adding 3 ml of the following salt solution to 20 ml of 10 percent water emulsion of the fatliquors taken in a test tube:

i) Calcium chloride solution	5 percent by mass
ii) Potash alum solution	5 percent by mass
iii) Sulphited quebracho	20 percent by mass
iv) Hydrochloric acid	5 percent by mass
v) Chrome liquor	5 percent by mass

A-1.2 If the emulsions are stable without creaming for a duration of at least 15 minutes *Min*, the product is stated to be satisfactory.

A-2 DETERMINATION OF *p*H

A-2.1 Determine the *p*H using *p*H-meter with a glass electrode in accordance with IS 5741 : 1970.

A-3 DETERMINATION OF WATER CONTENT

A-3.1 Outline of the Method

The percentage of water present is determined by distillation with a solvent.

A-3.2 Procedure

Heat 30 g of the product and 70 ml of a solvent (a light petroleum fraction, boiling between 100°C and 140°C is suitable) in a 300 ml flask connected, preferably by ground glass connections, to a condenser and a Dean and Stark receiving tube or similar apparatus graduated in divisions of 0·1 ml. Add a few glass beads to the flask to prevent bumping, but conduct the distillation in such a manner that the contents of the flask are in vigorous movement.

Continue the distillation until the liquid falling from the condenser is clear. Then disconnect the receiving tube, allow to stand and read off the volume of water which has separated from the solvent.

NOTES

1 Thorough cleaning of the apparatus by a mixture of chromic acid and sulphuric acid, followed by thorough washing with water prevents the formation of troublesome drops of water difficult to get into the measuring tube.

2 This method is not accurate if the sulphated oil contains mineral acids, free sulphonic acids or free sulphonic esters, alcohol, acetone or other water miscible solvents.

A-3.3 Calculation

Calculate the percentage of water present in the sample from the reading and the mass of the oil taken for the test.

A-4 DETERMINATION OF TOTAL FATTY MATTER

Follow the procedure given in Method A or Method B.

A-4.1 Procedure

A-4.1.1 *Method A*

If inorganic salts or soaps are present, heat 3·5 g of the sample with 10 ml of 25 percent hydrochloric acid under a reflux condenser until the fat separates out clearly and free from emulsion. Cool and add 50 ml diethyl ether. If after a brief shaking, the upper ethereal layer is not clear, repeat the operation with longer heating with hydrochloric acid.

Run the solution of fat and the acid wash-water into a separating funnel, run off the acid aqueous layer and when the ether layer is clear, wash with 10-percent sodium chloride (NaCl) until free from acid. If water soluble fatty acids are present, extract the combined aqueous layers again with ether. If other substances which are split by hydrochloric acid (HCl) only with difficulty are present (alkaline earth soaps, filling materials, etc), repeat the treatment with hydrochloric acid. If emulsions form, which prevent sharp separation, remove the material responsible for the formation of the emulsions by filtration and wash with ether. Dry the combine ether solutions for half an hour anhydrous sodium sulphate, having first rendered the sodium sulphate fat free by washing with dry ether. Filter the dried ethereal solution of the fat into a weighed flask. Distill off the greater portion of the ether and remove the final traces by blowing over the surface, preferably with a hand blower. The solvent rapidly evaporates and constant weight is rapidly attained by heating at 100°C. The weight may be taken as constant if it falls by not more than 0·1 percent during 15 minutes heating.

A-4.1.2 *Method B*

Where it is desired to extract the fat without pretreatment with hydrochloric acid, dissolve 3·5 g of the sample in 10 ml diethylether. Dry the ethereal solution over fat-free anhydrous sodium sulphate in a ground glass stoppered flask, to facilitate shaking, for 30 minutes and filter. If the ether insolubles are required, filter the ethereal solution before drying. Then follow the procedure given in **A-4.1.1**.

NOTE — The ether extract contains the saponifiable fatty matter (neutral fat and free fatty acids) and the other ether soluble materials, such as natural and added unsaponifiable matter, resins, naphthenic acid, and fatty acids combined with bases carried through the ether in small amounts only.

A-5 DETERMINATION OF TOTAL SULPHUR

A-5.1 Principle

Total sulphur in synthetic fatliquors is estimated by oxidation of sulphur to sulphuric acid and then titrating them with barium nitrate.

A-5.2 Apparatus

A-5.2.1 *Conical Flask* — with ground glass neck, 250 ml capacity.

A-5.2.2 *Oxygen Flask* — 250 ml capacity.

A-5.2.3 *Platinum Rod*

A-5.2.4 *Burette*

A-5.3 Reagents

A-5.3.1 *Barium Nitrate* — approximately N, accurately standardized.

A-5.3.2 *Hydrogen Peroxide* — 1·5 percent solution.

A-5.3.3 *Oxygen*

A-5.3.4 *Isopropyl Alcohol*

A-5.3.5 *Thorin Indicator*

A-5.4 Procedure

Take 4 to 5 ml of 1·5 percent solution of hydrogen peroxide in an oxygen flask filled with oxygen. Take the sample, accurately weighed (10 mg to 30 mg) at the tip of a platinum rod and inside an ashless filter paper. Ignite the filter paper and immediately transfer to the oxygen flask. Shake the flask well in order to ensure complete oxidation of sulphur.

After an interval of ten minutes, transfer the solution to a conical flask. Wash the stopper and the oxygen flask thoroughly with isopropyl alcohol and transfer the washings into the conical flask.

Titrate the content of the conical flask with standard barium nitrate solution using thorin as indicator. The change of colour from yellow to pink marks the end point.

A-5.5 Calculation

$$\text{Total sulphur, in percent} = \frac{N \times V \times 32}{1\,000} \times \frac{100}{W}$$

where

V = volume in millilitres of barium nitrate in the titration of the sample,

N = normality of barium nitrate solution, and

W = weight in grams of the sample taken.

A-6 DETERMINATION OF TOTAL CHLORINE

A-6.1 Principle

Total chlorine in synthetic fatliquors is estimated by oxidation of chlorine to chloride and then titrating them with silver nitrate.

A-6.2 Apparatus

A-6.2.1 *Conical Flask* — with ground glass neck, 250 ml capacity

A-6.2.2 *Oxygen Flask* — 250 ml capacity.

A-6.2.3 *Platinum Rod*

A-6.2.4 *Burette*

A-6.3 Reagents

A-6.3.1 *Silver Nitrate* — approximately N, accurately standardized.

A-6.3.2 *Hydrogen Peroxide* — 1·5 percent solution.

A-6.3.3 *Oxygen*

A-6.3.4 *Nitric Acid Solution* — approximately N, accurately standardized.

A-6.3.5 *Phenolphthalein Indicator*

A-6.3.6 *Sodium Hydroxide* — approximately N, accurately standardized.

A-6.4 Procedure

Take 4 to 5 ml of 1·5 percent solution of hydrogen peroxide and 15 ml of N sodium hydroxide in an oxygen flask filled with oxygen. Take the sample, accurately weighed (10 mg to 30 mg) at the tip of a platinum rod and inside an ashless filter paper. Ignite the filter paper and immediately transfer to the oxygen flask. Shake the flask well in order to ensure complete oxidation of sulphur.

After an interval of ten minutes, when the fumes subside, transfer the solution to a conical flask and neutralize with N nitric acid solution using phenolphthalein as indicator.

Titrate the resultant solution of the conical flask with standard silver nitrate solution using phenolphthalein as indicator. The change of colour from yellow to red marks the end point.

A-6.5 Calculation

$$\text{Total sulphur, in percent} = \frac{N \times V \times 35{\cdot}45}{10\,W}$$

where

V = volume in millilitres of silver nitrate in the titration of the sample,

N = normality of silver nitrate solution, and

W = weight in grams of the sample taken.

A-7 DETERMINATION OF TOTAL ASH

A-7.1 Weigh 3 g of material in a platinum dish and heat gently until a charred residue remains. Extract this with hot water to remove soluble salts, filter through a low ash filter paper and wash through. Ash the filter paper and charred residue, moistening with ash-free hydrogen peroxide, if difficulty is experienced.

Allow the dish to cool, add the aqueous extract and evaporate on a steam bath. Finally complete the ashing at as low a temperature as possible. Cool the dish and weigh.

A-7.2 Calculation

$$\text{Total ash of the material} = \frac{B \times 100}{A}$$

where

B = mass in g of the ash (after subtracting the ash content of filter paper), and

A = mass in g of the sample taken for test.

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